Data in Brief 33 (2020) 106345

Contents lists available at ScienceDirect

Data in Brief

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Data Article

Electrochemical data on redox properties of human Cofilin-2 and its Mutant S3D

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ARTICLE INFO

Article history:
Received 21 August 2020
Revised 15 September 2020
Accepted 18 September 2020
Available online 24 September 2020

Keywords:
Human cofilin
Disulfide bridge
Cyclic voltammetry
Redox properties
Electrochemistry
Cysteine

ABSTRACT

The reported data are related to a research paper entitled "Phosphorylated cofilin-2 is more prone to oxidative modifications on Cys39 and favors amyloid fibril formation" [1]. Info about the formation and redox properties of the disulfide bridge of a protein is quite difficult to obtain and only in a few cases was it possible to observe a cyclic voltammetry (CV) signal [2,3]. Human cofilin-2 contains two cysteines (Cys39 and Cys80) which can be oxidized in suitable conditions and form a disulfide bridge [1]. For this purpose, CV measurements were carried out on human cofilin-2 WT and its mutant S3D immobilized on a gold electrode coated by an anionic self-assembled monolayer (SAM), after a pre-oxidation time which was fundamental for observing a CV signal relating to the oxidation/reduction process of the disulfide bridge of the proteins. The data include CV curves obtained with and without electrochemical

DOI of original article: 10.1016/j.redox.2020.101691
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https://doi.org/10.1016/j.dib.2020.106345
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pre-oxidation and after oxidation with H_2O_2. In addition, the plot of the cathodic peak current *vs.* electrochemical pre-oxidation time and the pH dependence of the formal potential (E°') are reported. The data obtained by CV measurements were used to determine the time required to form the disulfide bridge for the immobilized proteins and, consequently, to observe the CV signal, to calculate the E°' values and analyse the pH dependence of E°'. The electrochemical data were provided which will be useful for further electrochemical investigations regarding proteins bearing disulfide bridge(s) or cysteines prone to oxidation.

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Specifications Table

Subject	Chemistry
Specific subject area	Electrochemistry. Cyclic voltammetry (CV) of cofilin-2 immobilized on anionic SAM
Type of data	Figure: CV curves Plot of E°' as a function of temperature Table: E°' as a function of temperature E°' as a function of pH Current as a function of scan rate Current as a function of pre-oxidation time
How data were acquired	Potentiostat/Galvanostat mod. 273A (EG&G PAR, Oak Ridge, USA)
Data format	Raw Analyzed
Parameters for data collection	The electrochemical experiments were carried out on cofilin-2 WT and S3D immobilized onto a SAM-coated Au electrode, functionalized with a mixed 11-Mercapto-1-undecanoic acid (MUA) and 11-Mercapto-1-undecanol (MU) SAM. The CV measurements were carried out at different scan rates (0.02–5 V s^{-1}) using a cell for small volume samples (0.5 mL) under argon atmosphere. A 1 mm-diameter polycrystalline gold wire, a Pt sheet, and a saturated calomel electrode (SCE) were used as working, counter, and reference electrode, respectively. The electrical contact between the SCE and the working solution was achieved with a Vycor® (from PAR) set.
Description of data collection	CV curves of cofilin-2 WT and S3D after electrochemical pre-oxidation CV curves of cofilin-2 WT and S3D after oxidation with H_2O_2 Standard potentials E°' vs. temperature and pH Cathodic currents vs. scan rate
Data source location	Department of Chemical and Geological Sciences, University of Modena and Reggio Emilia, Modena, Italy
Data accessibility	With the article (raw data in Supplementary materials)
Related research article	Marcello Pignataro, Giulia Di Rocco, Lidia Lancellotti, Fabrizio Bernini, Khaushik Subramanian, Elena Castellini, Carlo Augusto Bortolotti, Daniele Malferrari, Daniele Moro, Giovanni Valdrè, Marco Borsari, Federica del Monte, Phosphorylated cofilin-2 is more prone to oxidative modifications on Cys39 and favors amyloid fibril formation, *Redox Biol.* **37**, 2020, Article n.101691

Value of the Data

- Cyclic voltammograms allow the characterization of the redox properties of the disulfide bridge in cofilin-2.
- The data provide info useful for the study of the electrochemistry of disulfide bridge in other proteins or peptides.
- The data are useful to investigate the conditions leading to the oxidation of cysteines and the formation of intra- and inter-molecular disulfide bridges in proteins which in turn can induce unfolding effects.
- Intra- and inter-molecular disulfide bridges in proteins can arise from oxidative stress phenomena and can impact with the protein functionality.

1. Data Description

In this report, CV curves and redox data of human cofilin-2 WT and its phosphomimetic mutant S3D are presented. Phosphorylated (unphosphorylated) cofilin-2 accomplishes depolymerization (polymerization), severing and treadmilling of actin filaments in the skeletal muscle and in the heart. When immobilized on anionic SAM of 1-mercaptoundecanoic acid/1-mercaptoundecanol (MUA/MU), both WT and S3D can be electrochemically oxidized resulting in the formation of a disulfide bond between Cys39 and Cys80. The phosphomimetic mutant of cofilin-2 is oxidized more easily. The CVs of WT and S3D, recorded at a scan rate of 0.05 Vs^{-1} and subjected to a electrochemical pre-oxidation time of 50 s, are shown in Fig. 1 and 2. Both species show a well-defined reduction signal. Without electrochemical pre-oxidation, but after

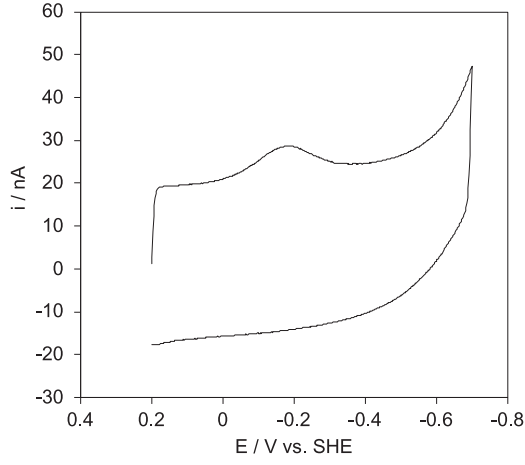

Fig. 1. Cyclic voltammogram of human cofilin-2 WT at scan rate 50 mV s^{-1} immobilized on Au wire coated by anionic SAM of MUA/MU. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. T = 293 K. Data points in Supplementary materials (SM1 data points Fig 1).

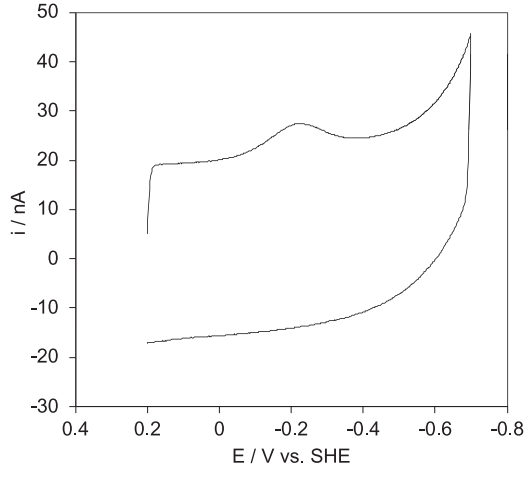

Fig. 2. Cyclic voltammogram of human cofilin-2 S3D at scan rate 50 mV s^{-1} immobilized on Au wire coated by anionic SAM of MUA/MU. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. T 293 K. Data points in Supplementary materials (SM2 data points Fig 2).

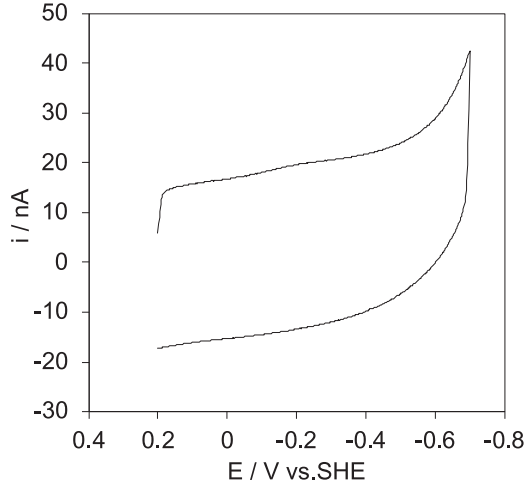

Fig. 3. Cyclic voltammogram of human cofilin-2 WT at scan rate 50 mV s^{-1} treated with 50 μm H_2O_2 for 1 h and immobilized on Au wire coated by anionic SAM of MUA/MU. Delay time: 0 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. T = 293 K. Data points in Supplementary materials (SM3 data points Fig 4).

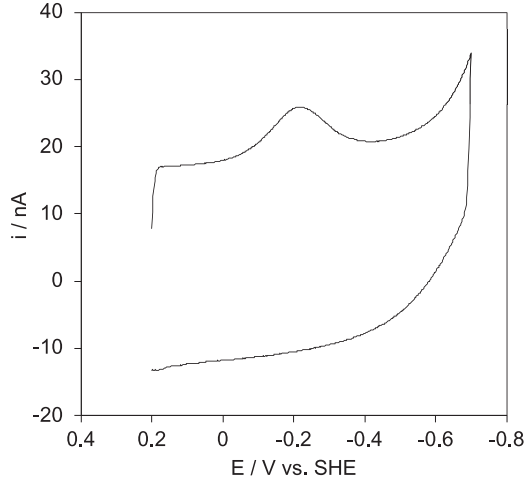

Fig. 4. Cyclic voltammogram of human cofilin-2 S3D at scan rate 50 mV s^{-1} treated with 50 μm H_2O_2 for 1 h and immobilized on Au wire coated by anionic SAM of MUA/MU. Delay time: 0 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. T = 293 K. Data points in Supplementary materials (SM4 data points Fig 4).

treatment with H_2O_2 (50 μM for 1 h), only S3D shows a reduction signal (Figs. 3 and 4). Plots of $E^{\circ\prime}$ *vs.* temperature for human cofilin-2 WT and S3D immobilized on anionic MUA/MU SAM are reported in Figs. 5 and 6, and the corresponding data are listed in Tab. 1. The E°' values of both proteins become more positive with increasing temperature. The pH dependent E°' values are reported in Tab. 2. Tab. 3 instead collects the cathodic peak currents vs. scan rate whose profile results linear, as expected for an adsorption controlled electron transfer process. The data presented here are related to the research article [1]. These redox data are of use for determining the electron transfer properties of disulfide bridges in proteins [2–4]. Disulfide/thiol reactions

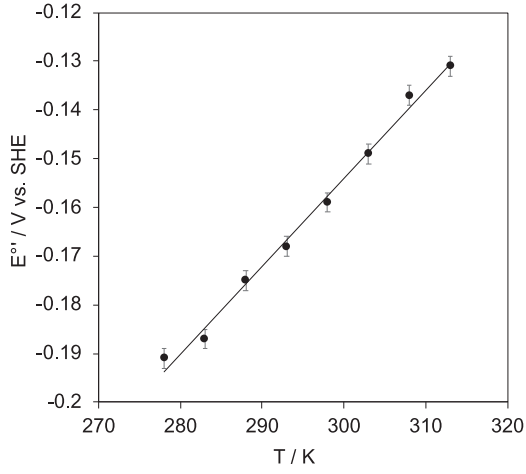

Fig. 5. Plot of E°′ *vs.* temperature for human cofilin-2 WT immobilized on anionic SAM of MUA/MU. Scan rate: 2 V $s^{-}1$. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. Data points of the corresponding CV in Supplementary materials (SM5 data points Fig 5).

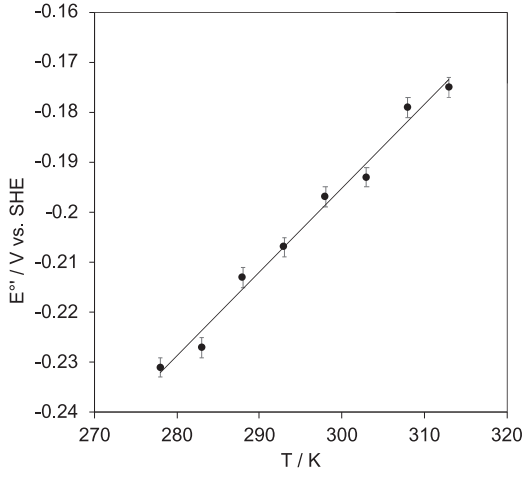

Fig. 6. Plot of E°′ versus temperature for human cofilin-2 S3D immobilized on anionic SAM of MUA/MU. Scan rate: 2 V $s^{-}1$. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. Data points of the corresponding CV in Supplementary materials (SM6 data points Fig 6).

are often critical in structural and conformational properties of proteins [5]. All electrochemical data (Fig. 1–6, Tab. 1–3) were obtained from cyclic voltammograms, run at scan rates from 0.02 Vs^{-1} to 5.00 Vs^{-1} [1]. All the data points and description of the CV curves (the plots I vs. E) used to obtain the data reported in Figures and Tables are available in Supplementary materials; the file name explicitly refers to the corresponding Figure or Table.

Table 1
E°' values at different temperatures for human cofilin-2 WT (CF-2 WT) and its variant S3D (CF-2 S3D) immobilized on anionic SAM of MUA/MU. Scan rate: 2 V s-1. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7. Error associated to E°' ±0.002 V. Data points of the corresponding CV in Supplementary materials (SM5 data points Fig 5; SM6 data points Fig 6).

T/K	CF-2 WT E°'/V	CF-2 S3D E°'/V
278	-0,191	-0,231
283	-0,187	-0,227
288	-0,175	-0,213
293	-0,168	-0,207
298	-0,159	-0,197
303	-0,149	-0,193
308	-0,137	-0,179
313	-0,131	-0,175

Table 2
E°' values at different pH values for human cofilin-2 WT (CF-2 WT) and its variants S3D (CF-2 S3D) immobilized on anionic SAM of MUA/MU. Scan rate: 2 V s-1. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate. T = 293 K. Error associated to E°' ±0.002 V. Data points of the corresponding CV in Supplementary materials (SM7 data points Tab 2 WT; SM7 data points Tab 2 S3D).

CF-2 WT pH	E°'/V	CF-2 S3D pH	E°'/V
4,92	-0,025	5,11	-0,075
5,87	-0,092	5,93	-0,127
6,76	-0,127	6,48	-0,148
7,31	-0,168	7,32	-0,207
8,11	-0,211	8,18	-0,249
8,89	-0,269	9,25	-0,326
9,23	-0,279	9,79	-0,349
9,87	-0,325		

Table 3
Cathodic peak currents at different scan rate values for human cofilin-2 WT (CF-2 WT) and its variants S3D (CF-2 S3D) immobilized on Au disk coated by anionic SAM of MUA/MU. Delay time: 50 s; Electrolyte solution: 10 mM sodium perchlorate and 5 mM buffer phosphate. T = 293 K, Ph = 7. Error associated to i ±5 %. Data points of the corresponding CV in Supplementary materials (SM8 data points Tab 3 WT; SM8 data points Tab 3 S3D).

scan rate/mV s^{-1}	CF-2 WT i/nA	CF-2 S3D i/nA
20	33	34
50	85	84
100	195	171
200	319	393
500	799	855
1000	1910	2310
2000	3398	3720
5000	8810	9650

2. Experimental Design, Materials and Methods

Cyclic voltammetry (CV) experiments were performed using a Potentiostat/Galvanostat PAR mod. 273A (EG&G PAR, Oak Ridge, USA). No signal was observed for human cofilin-2 WT and S3D in solution. For this reason, the electrochemical measurements were carried out on the proteins immobilized onto an Au electrode coated with a mixed 11-Mercapto-1-undecanoic acid (MUA) and 11-Mercapto-1-undecanol (MU) SAM which allowed to measure oxidation processes of cysteine residues with time. As control, the same measurements were repeated on the corresponding proteins alkylated at the sulfhydryl groups of the two cysteine residues present in the cofilin-2 WT and S3D. The oxidation of these cysteine residues cannot occur when they are alkylated and therefore they are electrochemically inactive. The sulfhydryl groups of the cysteine residues were derivatized by reaction with 15 mM iodoacetamide for 30 min at room temperature (20°C). The reactions were then quenched by adding an excess dithiothreitol (DTT). The achievement of the functionalization was verified through mass-spectrometry analyses performed on an ESI-Q-TOF accurate mass spectrometer (G6520AA, Agilent Technologies). In order to verify the efficiency of H_2O_2 as cysteine oxidant, electrochemical experiments were performed also on 5 μM cofilin-2 WT and S3D treated for 1 h with 50 μM H_2O_2 before adsorption on the SAM-coated Au electrode. The CV measurements were carried out at different scan rates (0.02–5 V s^{-1}) using a cell for small volume samples (0.5 mL) under Ar atmosphere [6]. A 1 mm-diameter polycrystalline gold wire, a Pt sheet, and a saturated calomel electrode (SCE) were used as working, counter and reference electrode, respectively. The electrical contact between the SCE and the working solution was obtained using a Vycor® (PAR) set. The cleaning of the working electrode is critical to obtain well-defined CV curves [6].

The working gold electrode was treated by flaming; afterwards, it was heated in concentrated KOH for 30 min, rinsed with water and subsequently cleaned by concentrated sulfuric acid for 30 min. To minimize residual adsorbed impurities, the electrode was subjected to 20 voltammetric cycles between +1.5 and -0.25 V (vs. SCE) at 0.1 V s^{-1} in 1 M H_2SO_4. Finally, the electrode was rinsed in water and anhydrous ethanol. The Vycor® set was treated in a water ultrasonic pool for about 5 min. SAM coatings on the gold electrode were obtained by dipping the polished electrode into a 1 mM ethanol solution of both MUA and MU for 12 h at 4°C and then rinsing it with MILLIQ water. Protein solutions were freshly prepared before use in 5 mM phosphate buffer at pH 7 and their concentration was carefully checked spectrophotometrically (Jasco mod. V-570 spectrophotometer). The immobilization of the proteins on the SAM-coated Au electrode was achieved by dipping the functionalized electrode into a 0.2 mM protein solution at 4°C for 5 h. 10 mM sodium perchlorate and 5 mM buffer phosphate at pH 7 were used as standard electrolyte solutions. Experiments at different pH values were performed as well, in this case the base electrolyte was 10 mM sodium perchlorate and 5 mM buffer phosphate and the pH were changed by adding small amount of concentrated NaOH or $HClO_4$. A well-shaped CV response on both human cofilin-2 WT and S3D was obtained by scanning the potential in the -0.2/+0.7 window, indicating a reversible oxidation/reduction process. A marked dependence on both the potential scan rate and the starting potential for the two proteins was, however, observed. In fact, both cathodic and anodic peaks were visible only when starting from an oxidative poise (*i.e.* applying a constant +0.2 V potential) with a delay time of at least 15 s and performing the CV at a scan rate of 1 Vs^{-1} or higher. On the contrary, at lower scan rates (0.02 Vs^{-1} < v < 0.5 Vs^{-1}) the CVs consisted of a cathodic peak only, and no voltammetric response could be obtained when starting from a reducing poise (-0.7 V vs. SHE), independently on the scan rate and on the delay time. When both the cathodic and anodic signals could be detected, the standard reduction potential E° was obtained as the semi-sum of the corresponding peak potential values. The formal reduction potentials E°' were found almost independent of scan rate in the range 0.02–5 V s^{-1}. The experiments were performed at least five times and the reduction potentials were found to be reproducible within ± 2 mV. As a negative control experiment, we performed electrochemical investigations using modified variants of WT and S3D cofilin-2,

previously treated with an alkylating agent and therefore featuring alkylated Cys. Under this condition, the proteins are no longer redox reactive and do not form the S-S bridge.

Supplementary Materials

Data points of all the CV curves used to obtain the data reported in the Figures and Tables.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

Acknowledgments

An appreciated support was provided by Centro Interdipartimentale Grandi Strumenti (CIGS) of Università di Modena e Reggio Emilia and its staff. This work was supported by the American Heart Association (grant AHA 14IRG18980028); University of Modena and Reggio Emilia (grant UNIMORE FAR2019); and MUSC (discretional funds).

Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:10.1016/j.dib.2020.106345.

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